

EVALUATION OF THE EFFECT OF SILICONE CONTAMINATION ON VARIOUS BOND SYSTEMS AND THE FEASIBILITY OF REMOVING THE CONTAMINATION

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Introduction

Silicone is a contaminant that can cause catastrophic failure of a bond system depending on the materials and processes used to fabricate the bond system. Unfortunately, more and more materials are fabricated using silicone. The purpose of this testing was to evaluate which bond systems are sensitive to silicone contamination and whether or not a cleaning process could be utilized to remove the silicone to bring the bond system performance back to baseline. Due to the extensive nature of the testing, attempts will be made to generalize the understanding within classes of substrates, bond systems, and surface preparation and cleaning methods.

This study was done by contaminating various metal (steel, Inconel, and aluminum), phenolic (carbon-cloth phenolic [CCP] and glass-cloth phenolic [GCP]), and rubber (natural rubber, asbestos-silicone dioxide filled natural butylidene rubber [ASNBR], silica-filled ethylene propylenediene monomer [SFEPDM], and carbon-filled ethylene propylenediene monomer [CFEPDM]) substrates which were then bonded using various adhesives and coatings (epoxy-based adhesives, paints, ablative compounds, and Chemlok® adhesives) to determine the effect silicone contamination has on a given bond system's performance. The test configurations depended on the bond system being evaluated. The study also evaluated the feasibility of removing the silicone contamination by cleaning the contaminated substrate prior to bonding. The cleaning processes also varied depending on bond system.

Experimental

The metal substrates were cleaned using an alkaline aqueous cleaning solution followed by a grit blast. The phenolic and rubber substrates were cleaned specific to the substrate material. The substrates were then contaminated with $10 \text{ mg}/\text{ft}^2 \pm 0.5 \text{ mg}/\text{ft}^2$ of silicone oil. The post-contamination cleaning process varied depending on the substrate being evaluated. Some samples were aged at 105 or $135 \pm 10^\circ \text{ F}$ and less than 50 percent relative humidity for 90 days. The lower temperature setting (105° F) was used for adhesives that have lower glass transition temperatures.

Results and Discussion

The bond systems were evaluated by testing six sample sets:

- Sample Set 1 (SS1) isolated the effect of the cleaning process on the adhesion properties (strength and failure mode) of the bond system. The test results of this sample set are considered baseline.
- Sample Set 2 (SS2) isolated the effect of accelerated aging on the baseline adhesion properties.
- Sample Set 3 (SS3) isolated the silicone contamination cleaning effectiveness of the process at $10 \text{ mg}/\text{ft}^2$.
- Sample Set 4 (SS4) isolated the effect of accelerated aging on the adhesion properties of contaminated and cleaned samples.
- Sample Set 5 (SS5) isolated the effect of $10 \text{ mg}/\text{ft}^2$ of silicone contamination on adhesion properties.
- Sample Set 6 (SS6) isolated the integrity of specimen preparation procedures and possibly differentiates anomalies in material used for sample assembly.

The bond systems with metal substrates evaluated were epoxy adhesive to D6AC steel, asbestos-filled epoxy adhesive to D6AC steel, epoxy adhesive to painted D6AC steel, and cork-filled ablative compound to painted aluminum. The specimens were cleaned using a combination of solvent wipe and hand abrade depending on the bond system being evaluated. Specimens were evaluated using a button-to-panel tensile strength configuration. The epoxy to D6AC steel was sensitive to silicone contamination and demonstrated a 75 percent decrease in tensile strength in the presence of $10 \text{ mg}/\text{ft}^2$ silicone contamination. The epoxy adhesive-to-D6AC steel bond system was cleaned using a trichloroethane (TCA) or PF Degreaser™ wipe. Even though the specimens that were cleaned showed an increase in strength when compared to the contaminated samples, the strength was approximately 50 percent lower than the baseline samples. The data are shown in Figure 1.

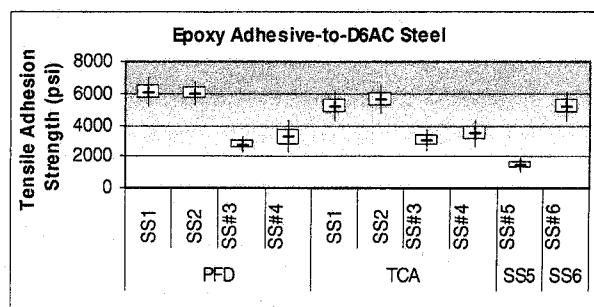


Figure 1. Epoxy Adhesive-to-D6AC Steel

The asbestos-filled epoxy adhesive to D6AC steel was sensitive to silicone contamination and demonstrated a 95

percent reduction in tensile strength in the presence of 10 mg/ft² silicone contamination. The asbestos-filled epoxy-adhesive-to-D6AC steel bond system was cleaned by abrading the surface followed by a Plus-4™ wipe. Even though the specimens that were cleaned showed an increase in strength when compared to the contaminated samples, the strength was approximately 55 percent lower than the baseline samples. There was an increase in tensile strength with aging of the samples. The 90-day aged baseline samples exhibited a 55 percent increase in strength in comparison to the 0-time baseline specimens. The specimens that were cleaned and aged for 90 days only demonstrated an 18 percent decrease in bond strength in comparison to the baseline. This is due to the adhesive having time to relax allowing for higher strength. The data are shown in Figure 2.

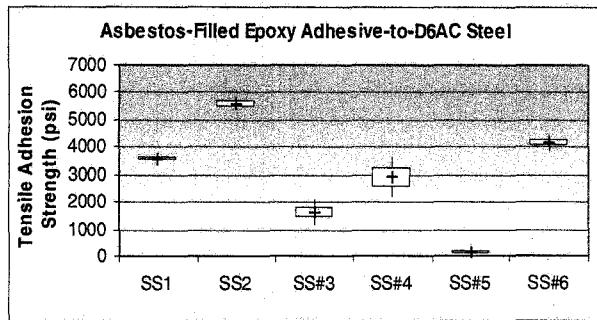


Figure 2. Asbestos-filled Epoxy Adhesive-to-D6AC Steel

The epoxy adhesive-to-painted D6AC steel and cork-filled ablative compound-to-painted aluminum were insensitive to silicone contamination. The bond systems demonstrated no decrease in tensile strength in the presence of 10 mg/ft² silicone contamination. Both bond systems were cleaned by abrading the surface followed by a PF Degreaser™ wipe. The asbestos-filled epoxy adhesive-to-ASNBR bond system was cleaned by a dry poly-wipe. The asbestos-filled epoxy adhesive-to-CFEPDM rubber bond system was cleaned by a PF Degreaser™ wipe followed by a dry poly-wipe. The data are shown in Figure 3 and Figure 4.

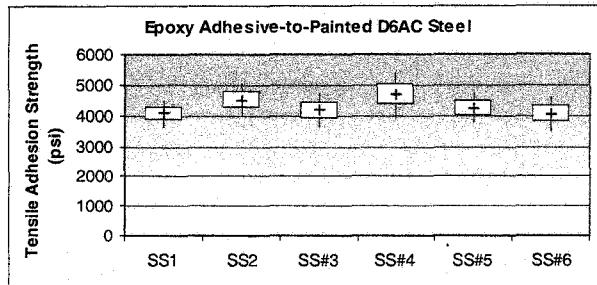


Figure 3. Epoxy Adhesive-to-Painted D6AC Steel

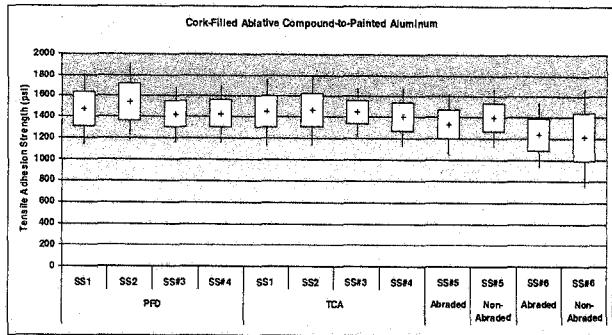


Figure 4. Cork-filled Ablative Compound-to-Painted Aluminum.

The bond systems with rubber substrates that have been evaluated are asbestos-filled epoxy adhesive-to-ASNBR, asbestos-filled epoxy adhesive-to-CFEPDM rubber, and cork-filled ablative compound-to-SFEPDM rubber. The specimens were evaluated using a button-to-panel tensile strength configuration. The asbestos-filled epoxy adhesive-to- ASNBR and asbestos-filled epoxy adhesive-to-CFEPDM rubber were insensitive to silicone contamination. The bond systems demonstrated no decrease in tensile strength in the presence of 10 mg/ft² silicone contamination. The asbestos-filled epoxy adhesive-to-CFEPDM rubber bond system was cleaned by a dry poly-wipe. The asbestos-filled epoxy adhesive-to-ASNBR bond system was cleaned by a PF Degreaser™ wipe followed by a dry poly-wipe. The data are shown in Figure 5 and Figure 6.

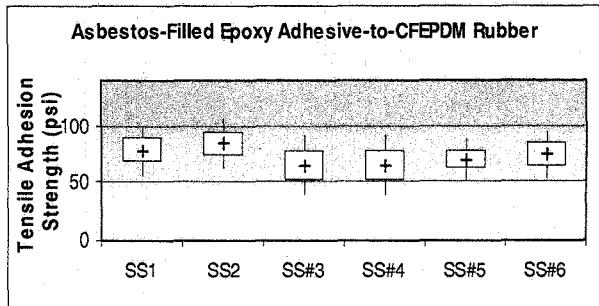


Figure 5. Asbestos-filled Epoxy Adhesive-to-CFEPDM Rubber

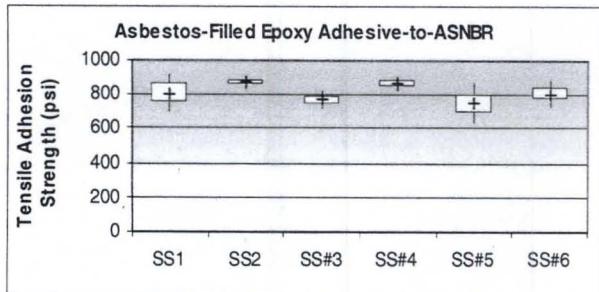


Figure 6. Asbestos-filled Epoxy Adhesive-to-ASNBR

The cork-filled ablative compound-to-SFEPDM rubber bond system was sensitive to silicone contamination and demonstrated a decrease of 35 percent in tensile strength in the presence of $10 \text{ mg}/\text{ft}^2$ silicone contamination. The cork-filled ablative compound-to-SFEPDM rubber bond system was cleaned using a PF Degreaser™ wipe. The specimens that were cleaned did not show an increase in strength when compared to the contaminated samples. The data are shown in Figure 7.

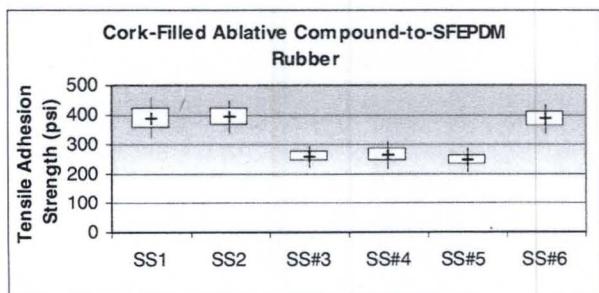


Figure 7. Cork-filled Ablative Compound-to-SFEPDM Rubber

The bond system with phenolic substrates that was evaluated was a pressure sensitive adhesive (PSA)-to-CCP. This bond system was evaluated by two test configurations: a button-to-panel tensile strength configuration and a T-peel peel strength configuration as shown in Figure 8. The pressure sensitive adhesive to CCP was insensitive to silicone contamination. The bond systems demonstrated no decrease in tensile strength in the presence of $10 \text{ mg}/\text{ft}^2$ silicone contamination. The PSA-to-CCP bond system was cleaned by abrading the surface followed by a TCA wipe. There was a decrease in strength due to 90-day aging but this phenomenon is inherent to the PSA and has been demonstrated in other testing. The data are shown in Figure 9 and Figure 10.



Figure 8. T-Peel Test Configuration

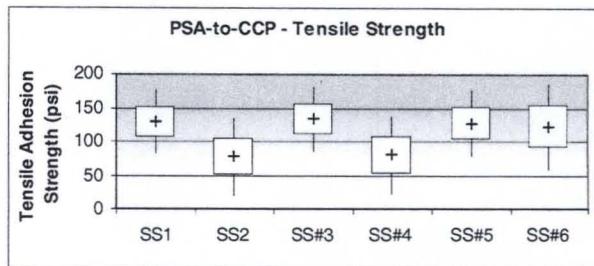


Figure 9. PSA-to-CCP Tensile Strength

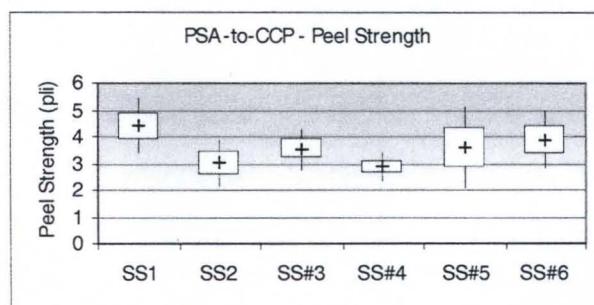


Figure 10. PSA-to-CCP Peel Strength

Conclusions

The filled and unfilled epoxy adhesive to unpainted metal and the cork-filled ablative compound to SFEPDM rubber have the most sensitivity to silicone contamination. The remaining bond systems that were evaluated did not demonstrate significant sensitivity to silicone. The testing demonstrated that if the bond system is sensitive to silicone contamination no simple means of cleaning method returned a bond to baseline. Further testing with the remaining substrates and adhesives and coating is still ongoing.

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Objectives



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- A. Determine the sensitivity of the various bond systems to a silicone contamination surface concentration of 10 mg/ft^2
- B. Determine if simple cleaning processes can sufficiently remove up to 10 mg/ft^2 of silicone contamination to achieve baseline adhesion properties (at zero time and after accelerated aging)

Background



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- **Silicone is a contaminant that can cause catastrophic failure of a bond system depending on the materials and processes used to fabricate the bond system**
- **As contamination detection technology improves, more and more materials are testing positive for silicone**

Table I. Master Matrix

Process Step	SS1	SS2	SS3	SS4	SS5	SS6
Fresh condition: •Metal substrates – grit blasted •Painted and nonmetal substrates – as cured then protected from other contaminants	X	X	X	X	X	X
Silicone contamination (10 mg/ft ²)			X	X	X	
Cleaning process	X	X	X	X		
Adhesive system application	X	X	X	X	X	X
Bond assembly	X	X	X	X	X	X
Aging at 105 or 135 ± 10 °F, ambient humidity, and 90 days		X		X		

Test Matrix (cont.)



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- Sample Set 1 (SS1) isolated the effect of the cleaning process on the adhesion properties (strength and failure mode) of the bond system. The test results of this sample set are considered baseline.
- Sample Set 2 (SS2) isolated the effect of accelerated aging on the baseline adhesion properties.
- Sample Set 3 (SS3) isolated the silicone contamination cleaning effectiveness of the process at 10 mg/ft².
- Sample Set 4 (SS4) isolated the effect of accelerated aging on the adhesion properties of contaminated and cleaned samples.
- Sample Set 5 (SS5) isolated the effect of 10 mg/ft² of silicone contamination on adhesion properties.
- Sample Set 6 (SS6) isolated the integrity of specimen preparation procedures and possibly differentiated anomalies in material used for sample assembly.

Materials Evaluated



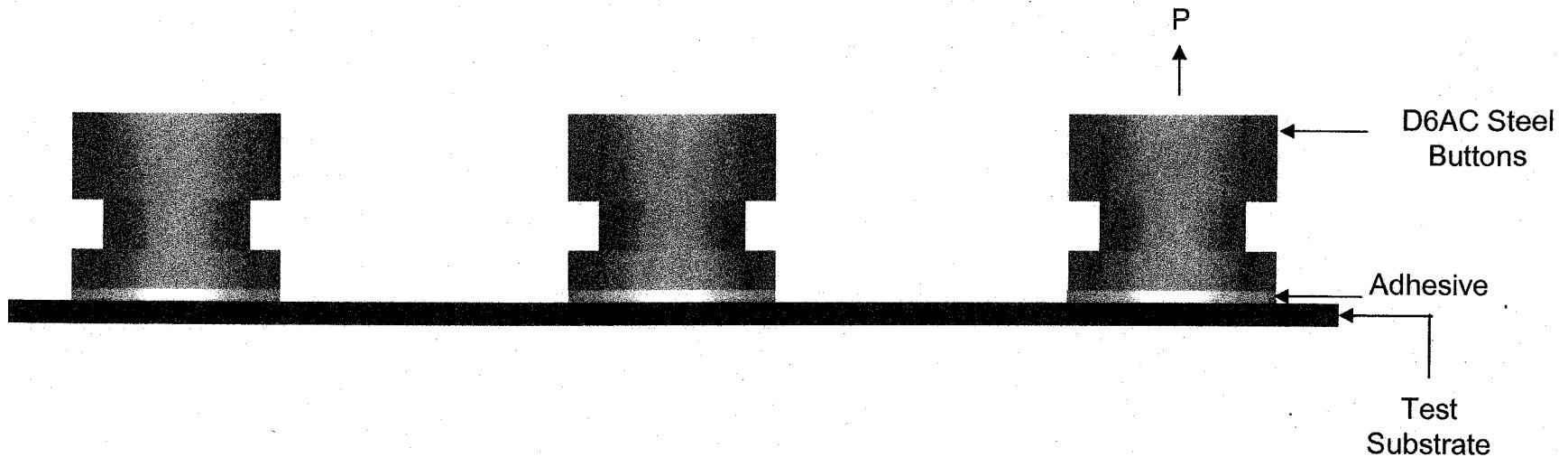
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- Substrates
 - Metals
 - D6AC steel
 - Aluminum
 - Painted D6AC steel
 - Painted aluminum
 - Rubber
 - Asbestos-silicone dioxide filled natural butyldiene rubber (ASNBR)
 - Silica-filled ethylene propylenediene monomer (SFEPM) rubber
 - Carbon-filled ethylene propylenediene monomer (CFEPDM) rubber
 - Phenolics
 - Carbon cloth phenolic (CCP)
- Adhesives
 - Epoxy adhesive
 - Asbestos-filled epoxy adhesive
 - Cork-filled ablative compound
 - Pressure sensitive adhesive
- Cleaning processes
 - Trichloroethane (TCA)
 - PF degreaser
 - Plus-4
 - Dry poly-wipe
 - Hand abrading

Test Configurations – Button to Panel



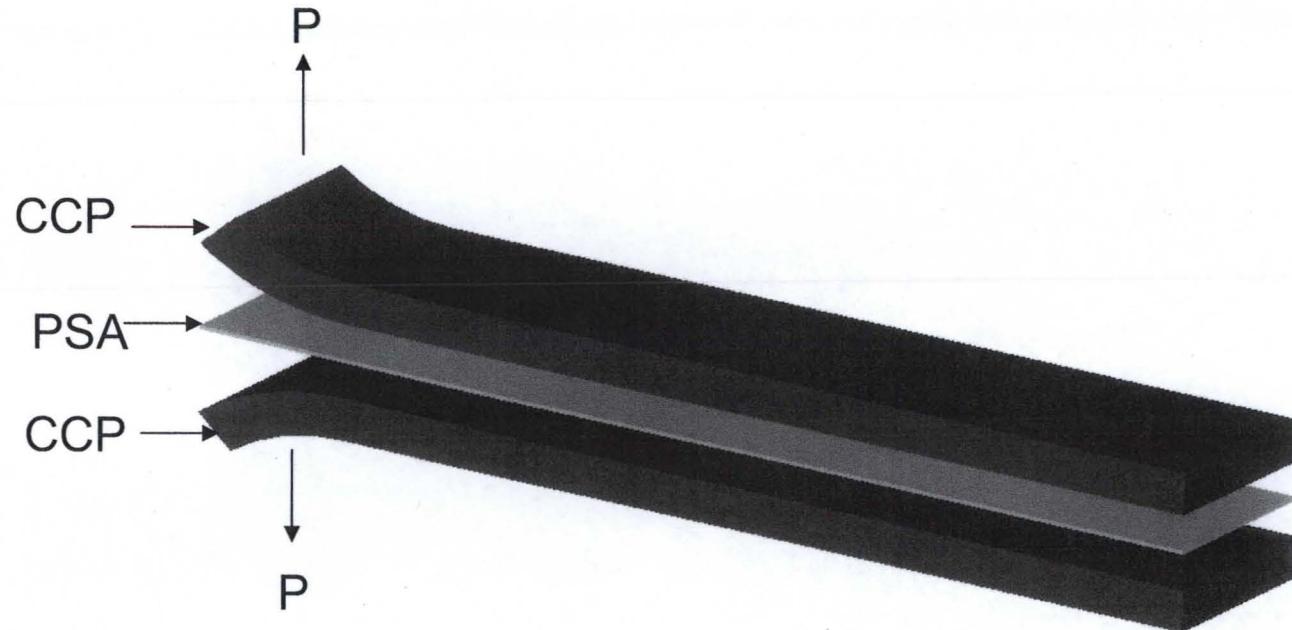
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Test Configurations – T-Peel



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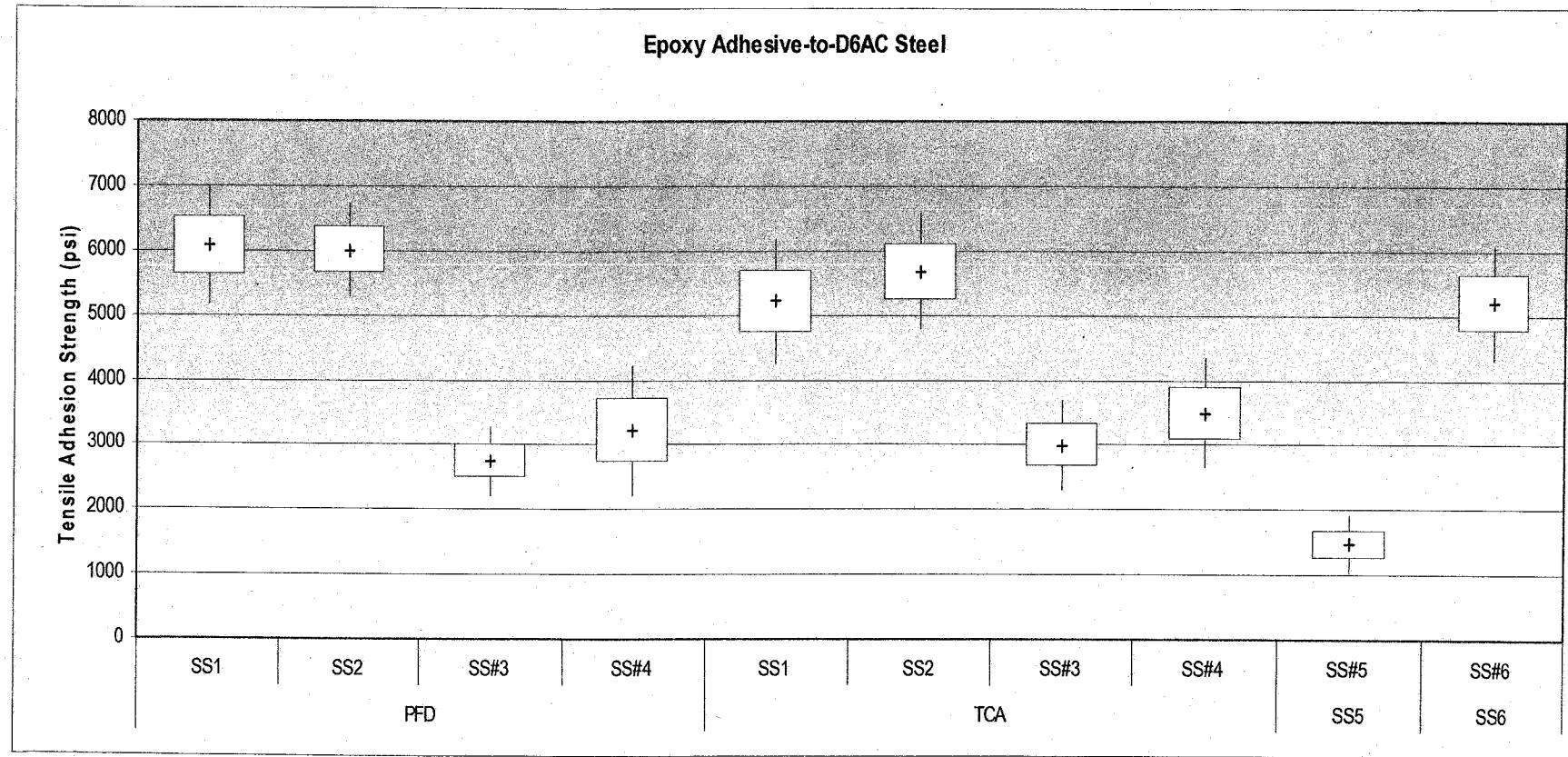


Epoxy Adhesive-to-D6AC Steel



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Cleaning Process – PF Degreaser™ Wipe or TCA Wipe



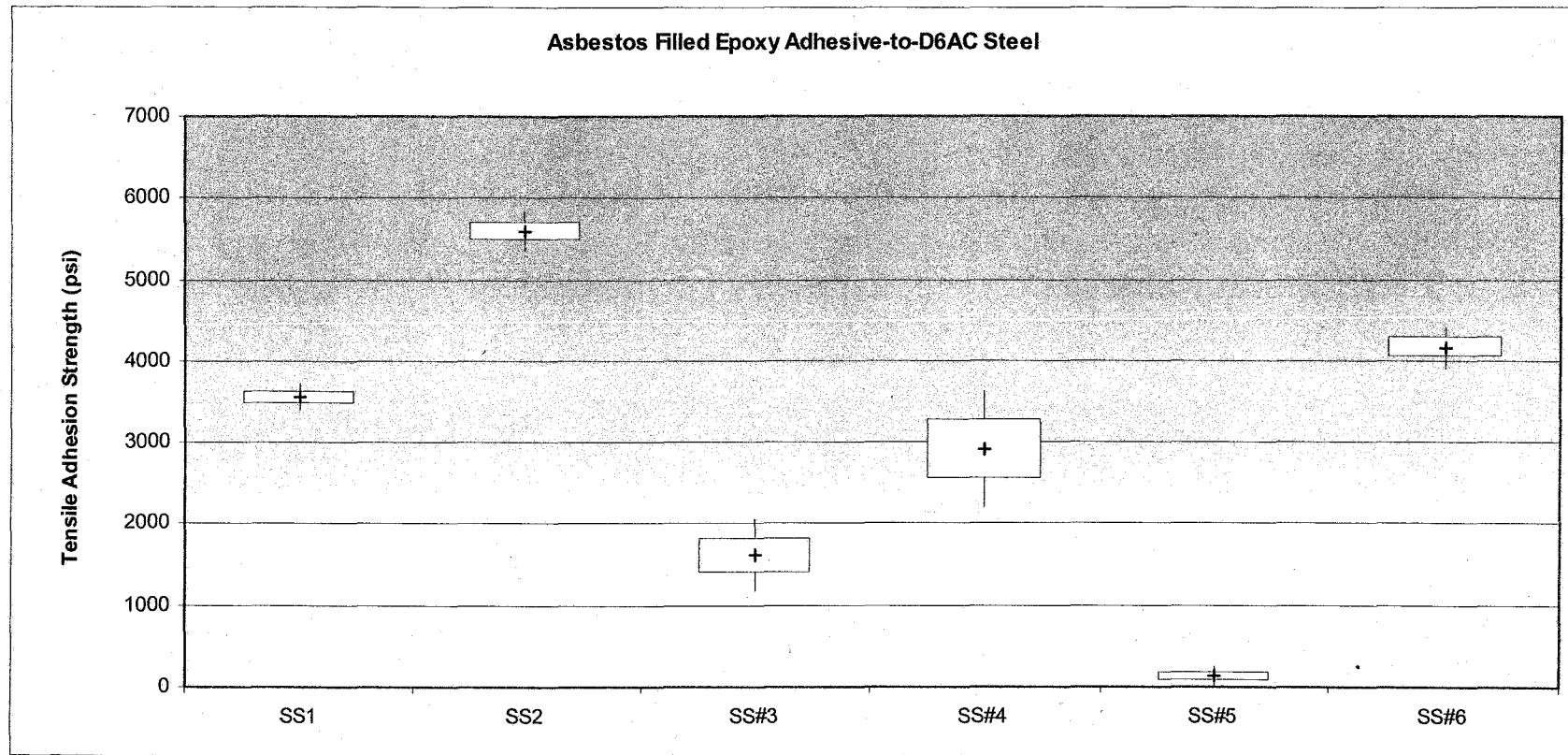
Conclusions: The epoxy adhesive-to-D6AC steel was sensitive to silicone contamination.

Asbestos-Filled Epoxy Adhesive-to-D6AC Steel



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Cleaning Process– Hand Abrade Followed By A Double Wipe With Pre-moistened
Plus-4™ Wipes



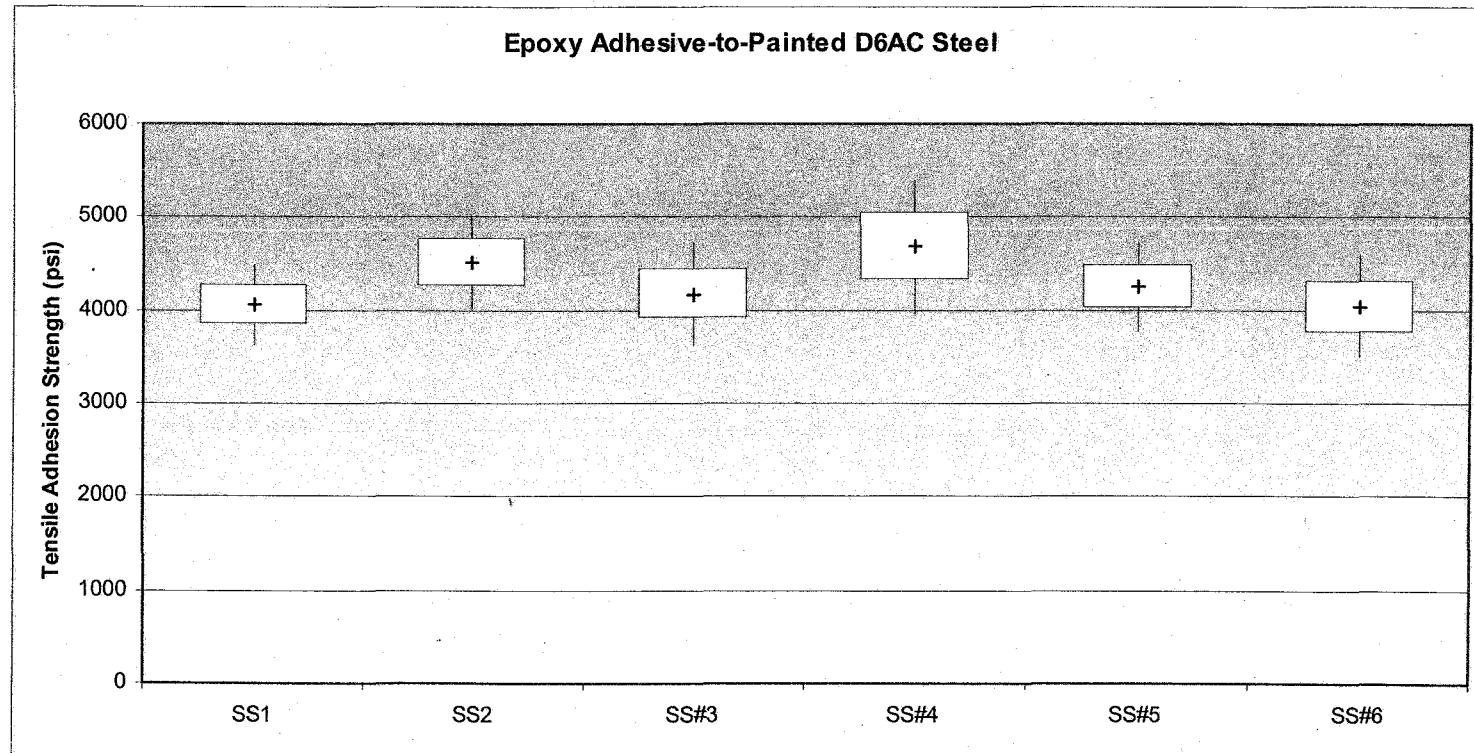
Conclusions: The asbestos-filled epoxy adhesive-to-D6AC steel was sensitive to silicone contamination.

Epoxy Adhesive-to-Painted D6AC Steel



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Cleaning Process – Abrade Followed by a PF Degreaser™ Wipe



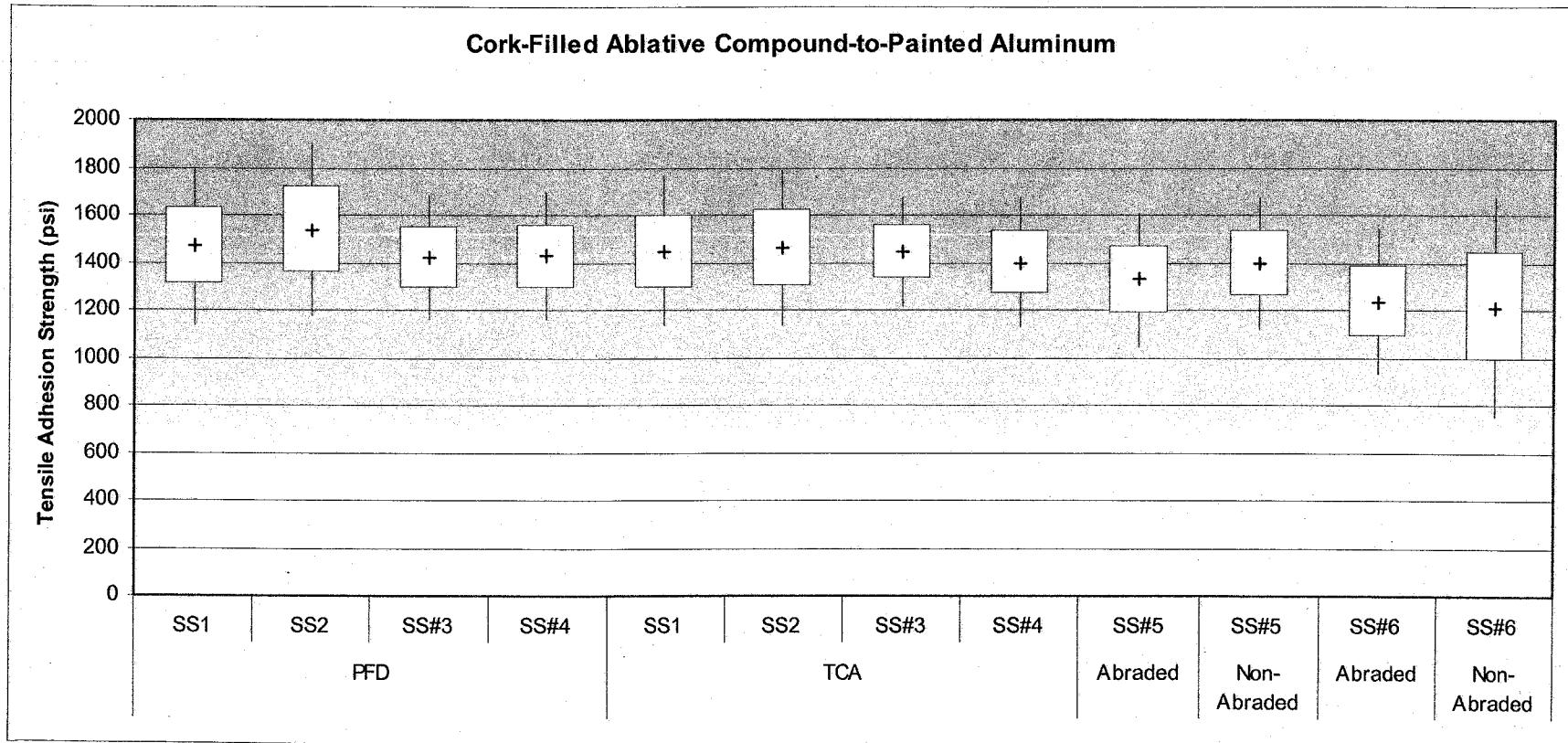
Conclusions: The epoxy adhesive-to-painted D6AC steel was insensitive to silicone contamination.

Cork-Filled Ablative Compound-to-Painted Aluminum



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Cleaning Process – Abrade Followed by a PF Degreaser™ Wipe

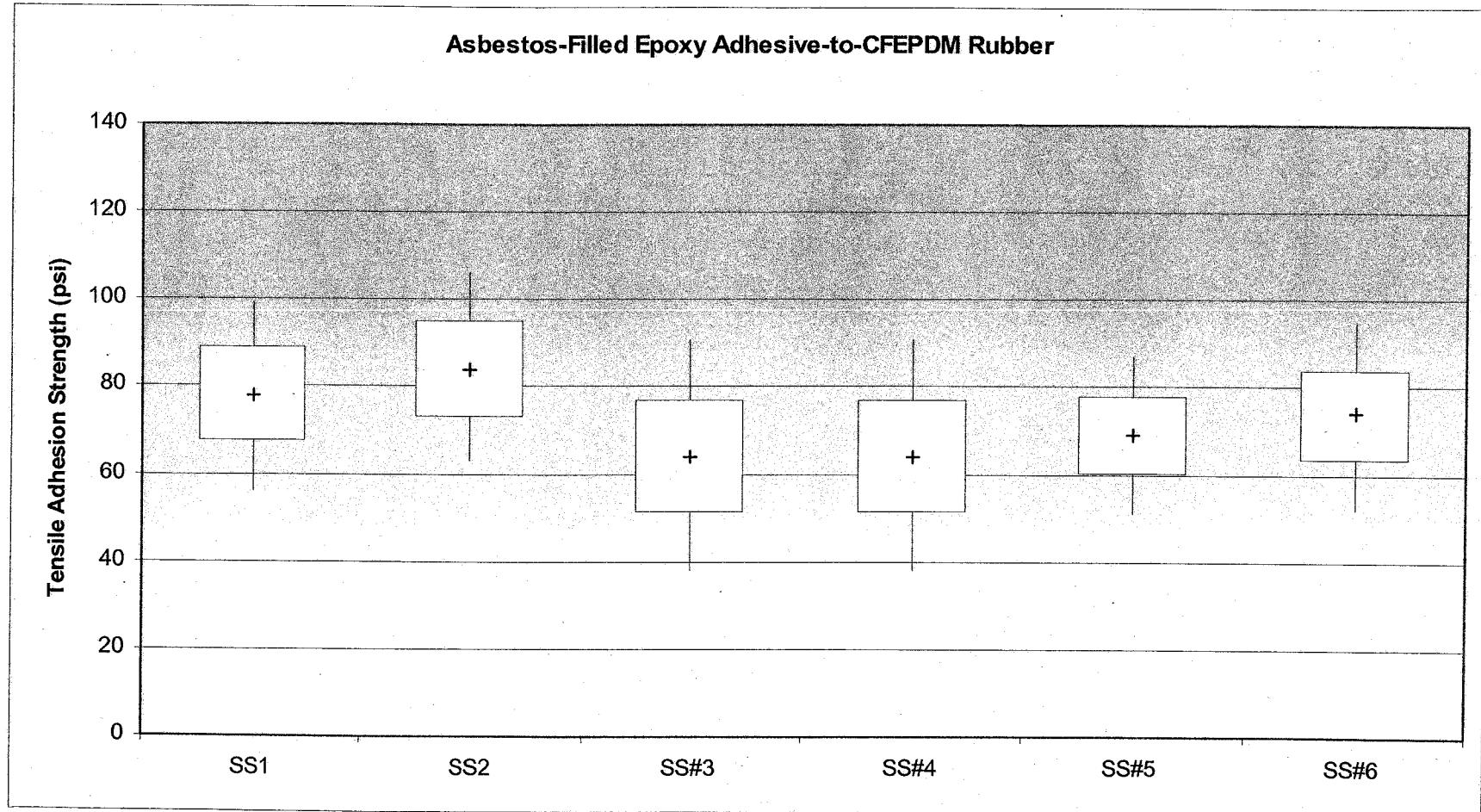


Conclusions: The cork-filled ablative compound-to-painted aluminum was insensitive to silicone contamination.

Asbestos-Filled Epoxy Adhesive-to-CFEPDM Rubber

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Cleaning Process – Wipe with a Dry Poly-wipe



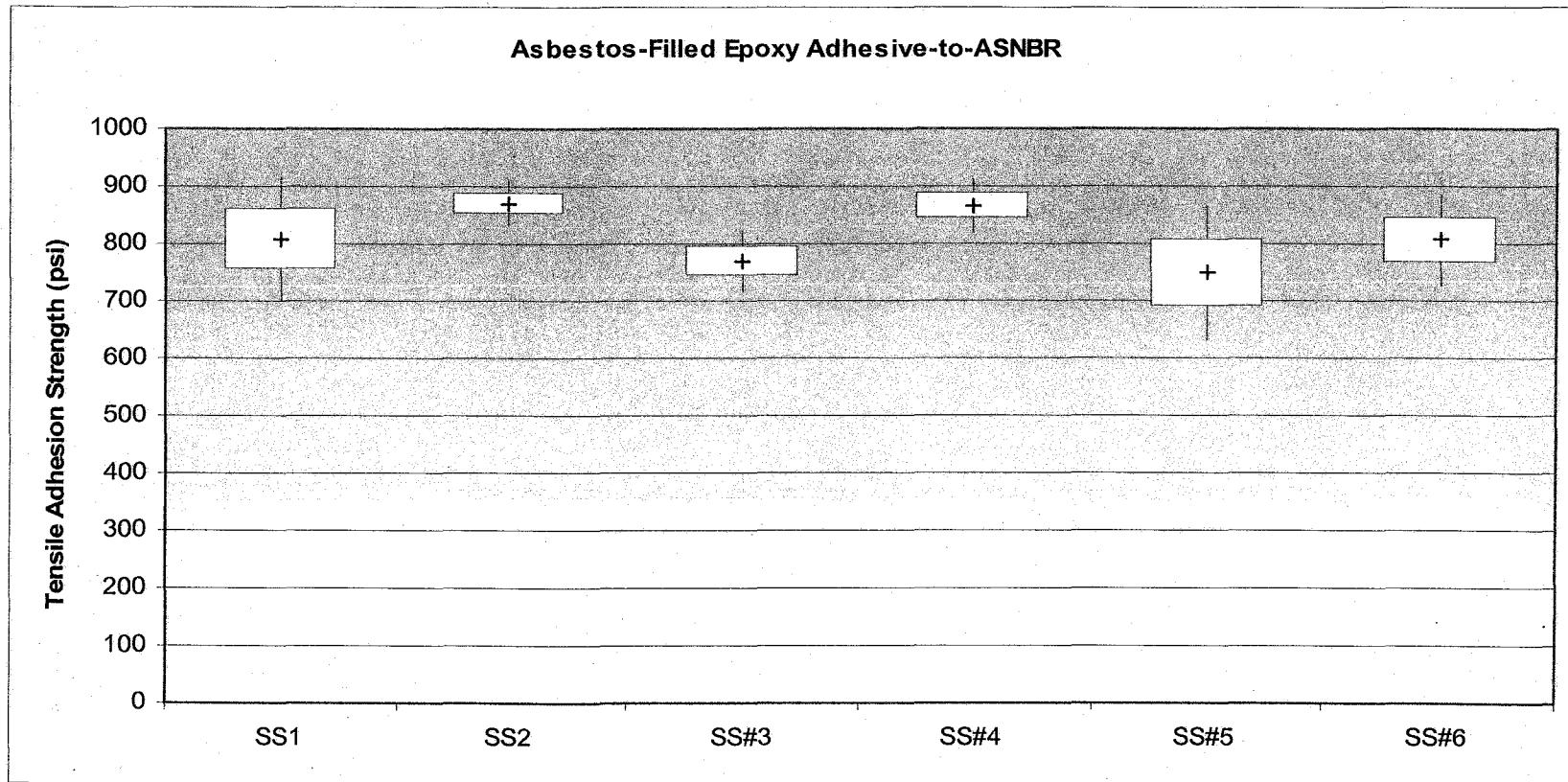
Conclusions: The asbestos-filled epoxy adhesive-to-CFEPDM rubber was insensitive to silicone contamination.

Asbestos-Filled Epoxy Adhesive-to-ASNBR



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Cleaning Process – PF Degreaser™ Wipe Followed By A
Dry Poly-wipe



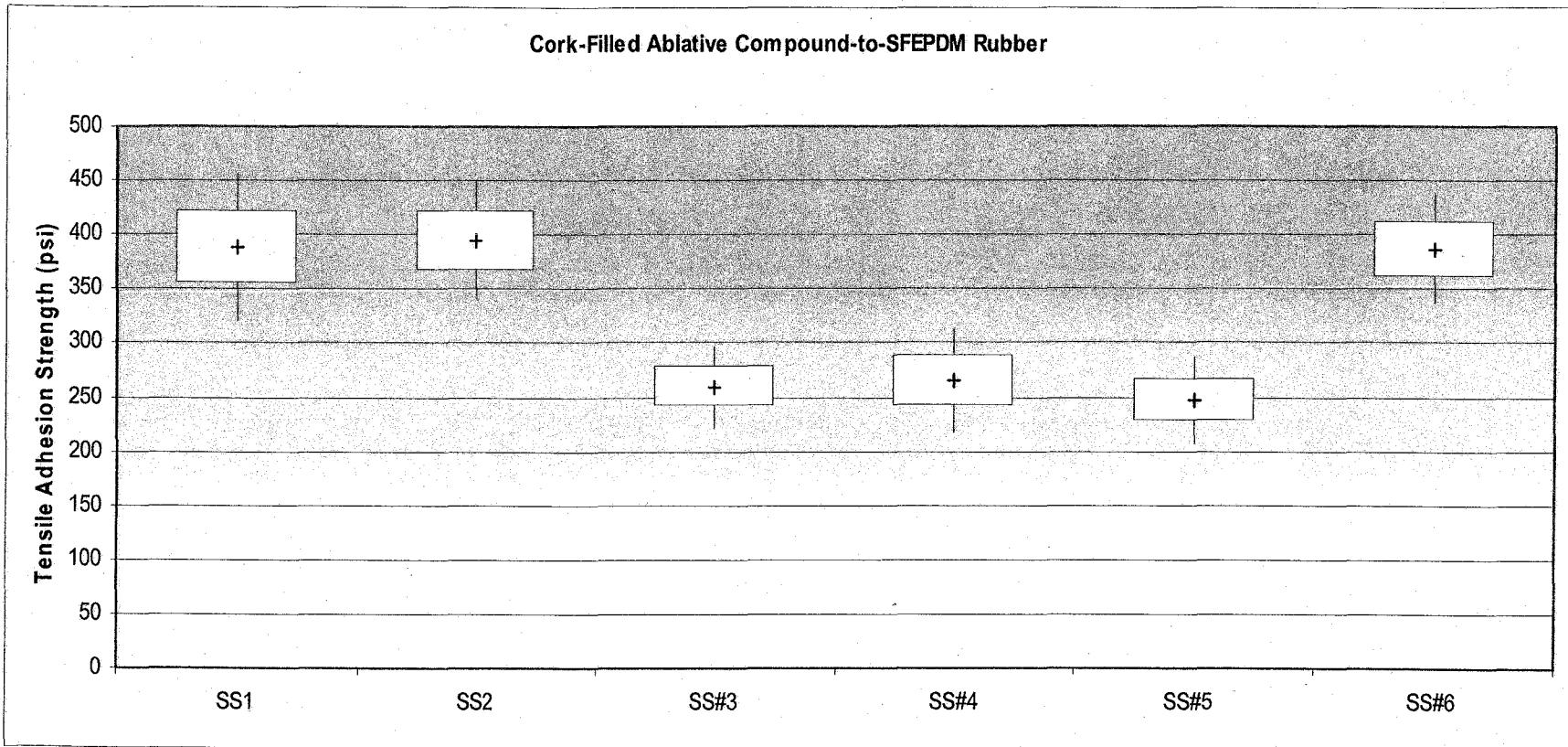
Conclusions: The asbestos-filled epoxy adhesive-to-ASNBR rubber was insensitive to silicone contamination.

Cork-Filled Ablative Compound-to-SFEPDM Rubber



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Cleaning Process – PF Degreaser™ Wipe



Conclusions: The cork-filled ablative compound-to-SFEPDM was sensitive to silicone contamination.

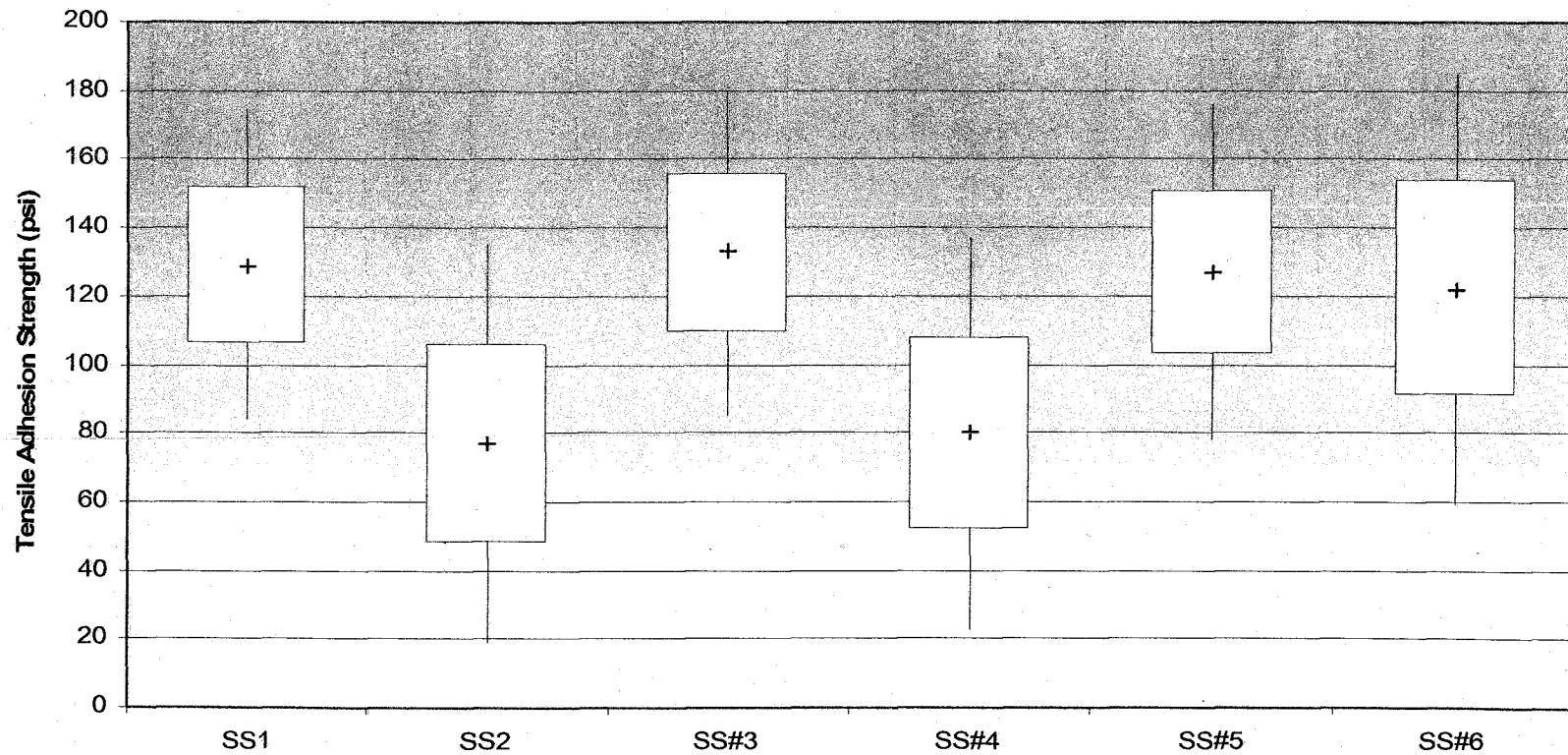
Pressure Sensitive Adhesive-to-CCP Button to Panel Test Configuration



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Cleaning Process – Abrade Followed by a TCA Wipe

Pressure Sensitive Adhesive-to-CCP - Button to Panel Test Configuration



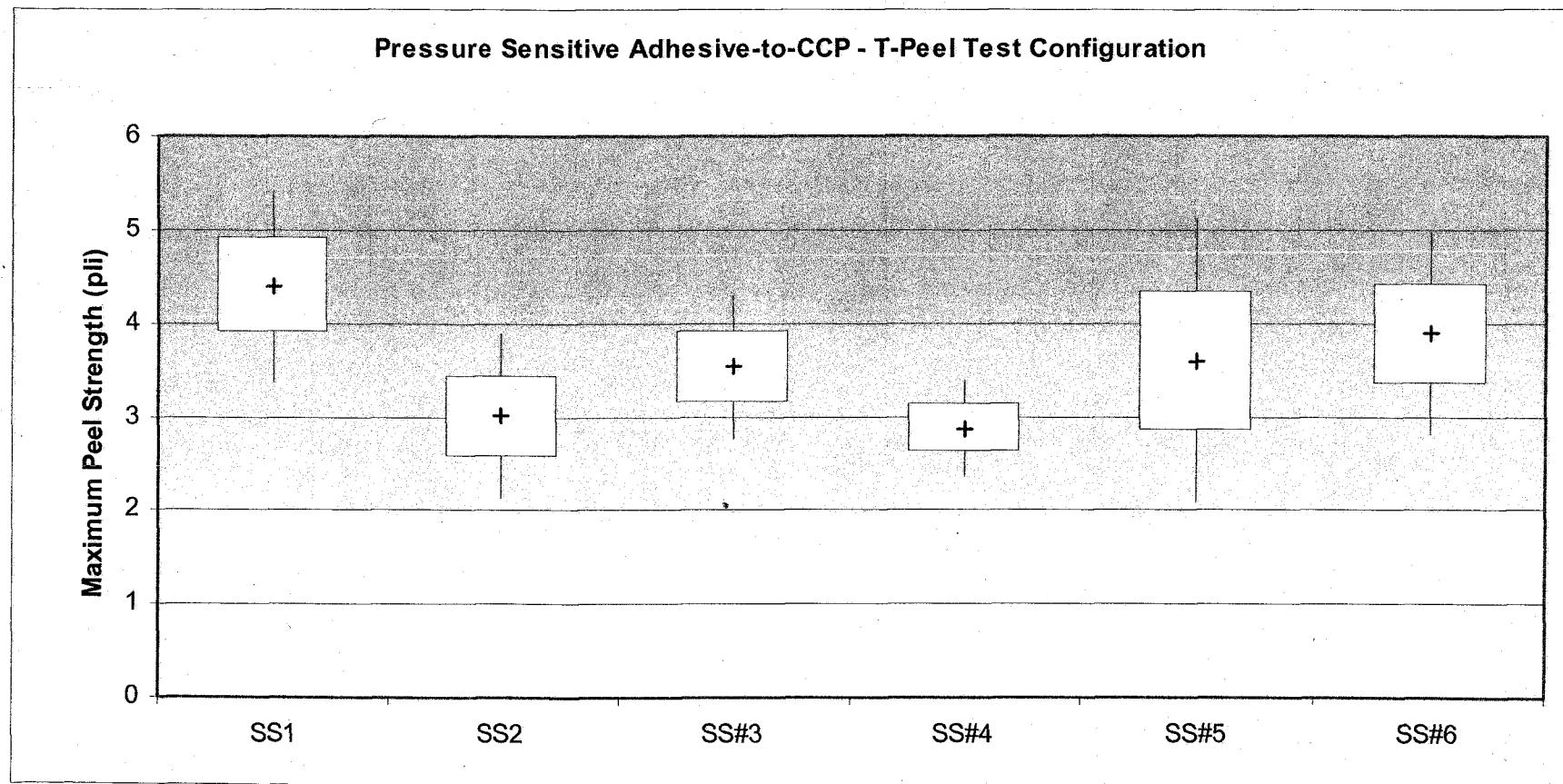
Conclusions: The pressure sensitive adhesive-to-CCP was insensitive to silicone contamination.

Pressure Sensitive Adhesive-to-CCP T-Peel Test Configuration



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Cleaning Process – Abrade Followed
by a TCA Wipe



Conclusions: The pressure sensitive adhesive-to-CCP was insensitive to silicone contamination.

Conclusions



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- The epoxy (filled and unfilled) adhesive-to-unpainted metal and cork-filled ablative compound-to-SFEPDM rubber have the most sensitivity to silicone contamination.
- The remaining bond systems that were evaluated did not demonstrate sensitivity to silicone.
- The research demonstrated that if the bond system was sensitive to silicone contamination simple cleaning methods would not return the bond performance to baseline.